

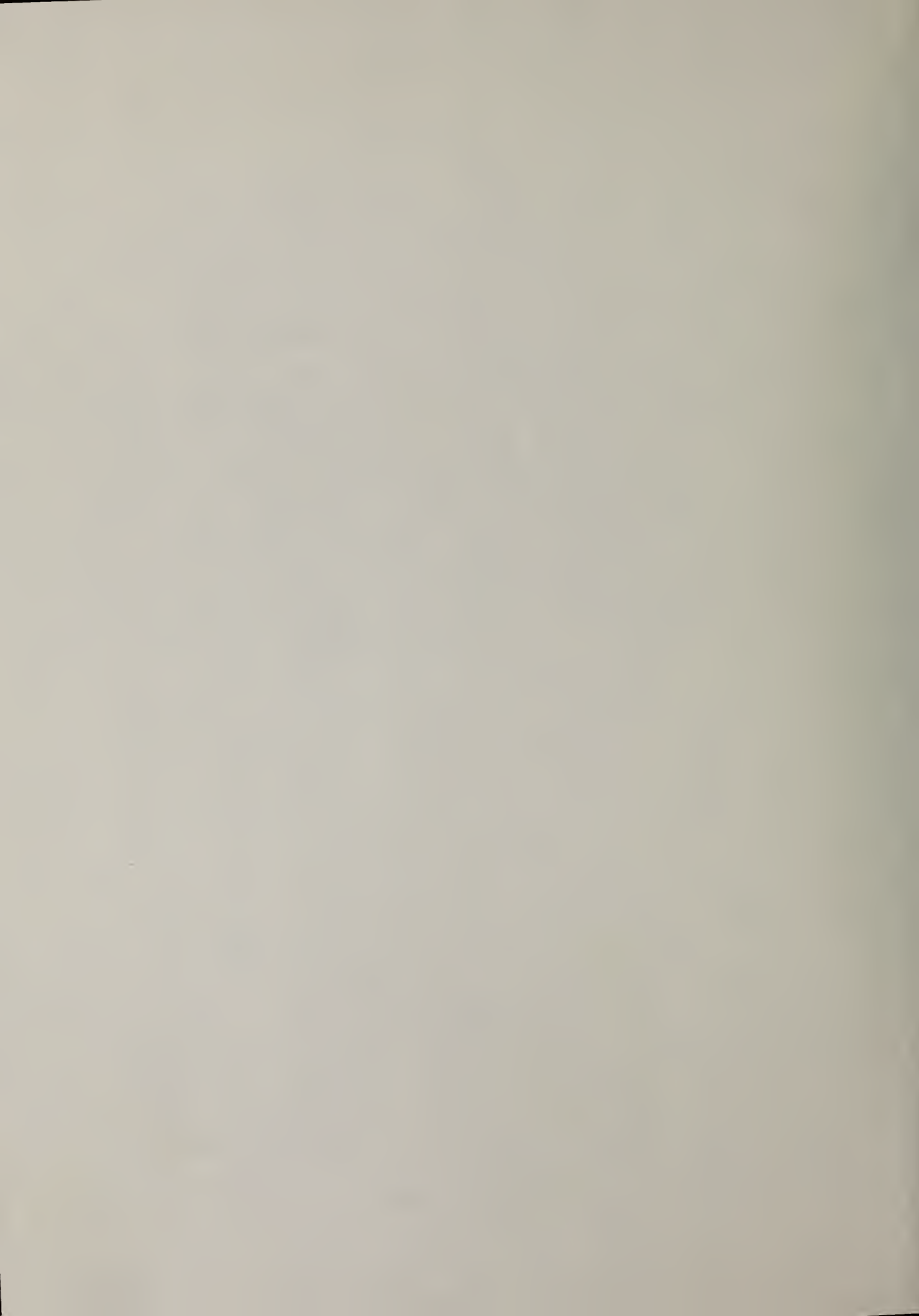
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Review of Material Provided by EPA on the Analysis for Organic Chemicals in the EPA Love Canal Monitoring Study

May 1982



U S DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS



NBSIR 82-2511

REVIEW OF MATERIAL PROVIDED BY
EPA ON THE ANALYSIS FOR ORGANIC
CHEMICALS IN THE EPA LOVE CANAL
MONITORING STUDY

May 1982

U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*



Review of Material Provided by EPA on the Analysis
for
Organic Chemicals in the EPA Love Canal Monitoring Study

NBS Review Panel

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FOREWORD

On July 20, 1981, EPA requested NBS to review the analysis for organic chemicals performed by EPA in its Love Canal Monitoring Study. In response to that request, this review has been prepared by a panel of NBS scientists with expertise in organic analytical chemistry, quality assurance and statistics. The review is based upon written material, provided by EPA to NBS, related to the acquisition of organic analytical data for samples collected from the Love Canal vicinity by EPA contractors.

The charge from EPA, accepted by NBS on August 17, 1981, was to carry out the following seven tasks:

1. Review the sample collection and analytical protocols for organic chemicals in air, water, soils, sediments and biota to determine their appropriateness for identifying and measuring the substances of interest to EPA at Love Canal.
2. Review the adequacy of the quality assurance/quality control protocols for all of the media in Task 1 to assure the validity of substance identification and analytical measurements.
3. Review the results of the quality assurance program presented in the EPA Love Canal report to assess the performance of the analytical program.
4. Review as necessary any of the reports of the on-site laboratory audits carried out by EPA.
5. Review the results of the EPA audit of GC-MS tapes to evaluate the quality of the analytical program.
6. Review any of the monitoring data as necessary to help in the overall review of the program.
7. Prepare a report on the overall adequacy of the EPA analytical and quality assurance protocols to meet the organic chemical monitoring goals (accuracy and precision) as set forth in the EPA Love Canal Monitoring Plan.

On August 18, 1981, NBS received an initial set of documents for review, and on August 25, NBS was given an oral briefing by officials of EPA and representatives of the prime contractor, the GCA Corporation, on the conduct of the Love Canal Monitoring Study. At the time NBS initiated its review, no final report on the Love Canal Monitoring Study was available. As a result of the unavailability of such a report, Tasks 3 and 7 were modified to:

3. Review the results of the quality assurance program as revealed in documents provided to NBS describing the performance of the analytical program.

and

7. Based upon written material provided by EPA, prepare a report summarizing the reviews conducted by NBS in Tasks 1 to 6, paying particular attention to conclusions drawn by EPA concerning the precision and accuracy of organic analytical data from the Love Canal Monitoring Study.

In order to give structure and specificity to the review, the NBS panel devised a set of questions to serve as guidance for the evaluation of the organic analytical portion of the EPA Love Canal Monitoring Study. This set of questions, designated in correspondence between EPA and NBS as the template for the review, was transmitted to EPA on October 13.

On December 9, 1981, NBS submitted its review of material received from EPA. That material consisted of a variety of documents including contractor plans, tables of data derived from samples taken from the Love Canal vicinity, tables of data derived from internal and external performance check samples, quality assurance audit reports, miscellaneous letters and reports, and a preliminary draft of portions of a final report. This latter document was designated as OEM-LC-2 by NBS. The December 9 review identified a number of deficiencies in the Love Canal Monitoring Study on the basis of the written material provided by EPA. In the cover letter to EPA accompanying the December 9 review, NBS stated that the responses to the template questions would be submitted to EPA in January 1982.

On December 15, EPA requested NBS to review additional material, and on December 17, EPA provided NBS with a copy, designated #9, of a draft report. NBS was informed that those sections of the draft report which dealt with the quality assurance aspects of the study were still in the process of being revised. NBS was requested to work with the revised versions. Since the additional material influenced the NBS responses to the template questions, NBS decided not to transmit the responses until the additional material had been reviewed. By February 17, 1982, the last of the additional material provided by EPA, which included revisions of the draft report and a copy of the final report of the prime contractor, was received. At the request of EPA, NBS briefed EPA on the contents of this review on April 15, 1982.

The present Review covers all material received, with the exception of the aforementioned OEM-LC-2, which was replaced by the draft report. All documents received by NBS are catalogued at the end of this Review.

Finally, the authors of this Review wish to acknowledge the advice and assistance of Prof. Michael Gross, Mr. Hugh Huffman, and Dr. James Sphon.

INTRODUCTION

This is a review by the National Bureau of Standards of the analysis for organic chemicals conducted by the U. S. Environmental Protection Agency in the Love Canal Area of Niagara Falls, New York. In conducting this review, NBS has been guided by the seven tasks specified by EPA in the charge given to NBS.

To give additional structure to the review and to ensure that all appropriate aspects of the Organic Analytical Program of the Love Canal Monitoring Study were considered, NBS devised a series of questions concerning various aspects of the program to serve as a detailed guide for the review. This set of questions has been designated in correspondence between NBS and EPA as the "template" for the review and the responses to the template questions are included as Appendix A of this Review.

In this Review, NBS has commented upon deficiencies or limitations that NBS has identified in the Love Canal Monitoring Study. Not all of these deficiencies or limitations influence the conclusions drawn by EPA in its draft report and some have already been described and interpreted by EPA. The purpose of including such comments is to call to the attention of readers particular limitations which might not be immediately apparent or which should be considered by those who wish to use the EPA data to draw independent conclusions. When deficiencies or limitations are identified which might affect the conclusions drawn by EPA in its draft report, they are so noted.

Finally, since the NBS review was to be concerned solely with the analysis for organic chemicals, other aspects of EPA's Love Canal Monitoring Study have been excluded, by agreement, from this review. Specifically, these are:

1. The choice of sample locations and media sampled. NBS did review, however, how the sample collection protocols might have affected the chemical integrity of the samples being collected.
2. The reduction of acquired data leading to contamination level summaries and distribution patterns. NBS did review, however, those statistical procedures associated with the determination of precision, accuracy, and limits of detection in the analysis of environmental samples for organic chemicals.
3. The choice of compounds which would be determined in the samples collected from the Love Canal vicinity. NBS did consider, however, the effect of selecting a list of targeted substances on the overall analytical program.
4. The analytical programs for inorganic chemicals and radioactivity.
5. The conclusions drawn by EPA from the Love Canal Monitoring Study. NBS did consider, however, whether or not the quality and reliability of the organic analytical data might affect the conclusions.

REVIEW OF THE ANALYSIS FOR ORGANIC CHEMICALS IN THE EPA LOVE CANAL MONITORING STUDY

EPA STUDY GOALS

The EPA Love Canal Monitoring Study was undertaken to accomplish three explicit goals:

1. Determine the current extent and degree of chemical contamination in the area defined by the emergency declaration order.
2. Assess the near-term and long-term implications of groundwater contamination in the general vicinity of Love Canal.
3. Provide an assessment (from an environmental contamination perspective) of the habitability of residences included in the emergency declaration order.

EPA STUDY DESIGN FOR THE ANALYSIS FOR ORGANIC CHEMICALS

The monitoring effort involved the analysis of environmental samples for more than 100 targeted substances. The substances targeted for analysis were selected from those known to have been buried in Love Canal, those previously observed in the environment near Love Canal, and the EPA list of priority pollutants. In addition to the targeted substances, the EPA draft report cites as one of the major safeguards in the monitoring study the identification of the 20 most abundant non-targeted substances in each environmental sample.

Four media--air, water, soil and sediments, and biota--were to be independently studied using gas chromatography-mass spectrometry (GC-MS) as the primary analytical technique. A detailed quality assurance program was designed for which the number of quality assurance samples to be analyzed equaled the number of field samples. From the analytical data, EPA was to generate a validated data base for the environmental samples.

SUMMARY OF THE NBS REVIEW

In response to a request from EPA, NBS has reviewed the analysis for organic chemicals in the EPA Love Canal Monitoring Study. The review is based upon written documentation provided to NBS during the period August 18, 1981 to February 17, 1982. (See Appendix B for the list of documents received.) The review was confined to those subjects discussed in the Foreword and the Introduction to this report.

- EPA's stated goals and objectives, as they applied to the analysis for organic chemicals, were appropriate in that media, substances, potential patterns of contamination, and temporal variation were considered. They were not, however, quantitatively explicit. EPA did not define at the outset of the study the limits of detection or the precision and accuracy required to permit comparison of levels of contamination in the Declaration Area with levels which constitute an environmental hazard or with levels currently found in U.S. cities.

In the implementation of the monitoring program, the requirement for the identification of the 20 most abundant non-targeted substances was generally not met. Consequently, the possibility of contamination by non-targeted substances has not been conclusively excluded. This deficiency does not invalidate, however, the investigation of targeted substances.

The quantitation limits realized by the water monitoring laboratories for individual analytes varied by more than a factor of ten. Few laboratories reported quantitation limits as good as those reported by the EPA laboratory responsible for the water monitoring effort. Comparisons of quantitation limits with data provided by EPA from other studies indicated that the contract laboratories were not uniformly performing at the state of the art in the water monitoring program.

In any study involving measurements, conclusions are bounded by measurement error and limits of detection. EPA has not fully used its quality assurance data to derive estimates of precision, accuracy, and limits of detection. EPA has not incorporated estimates of precision, accuracy, and limits of detection into its validated data base. Unless measured values, including "none detected," are accompanied by estimates of uncertainty, they are incomplete and of limited usefulness for further interpretation and for drawing conclusions.

SPECIFIC FINDINGS

In the remainder of this Review, specific comments are grouped according to the seven tasks in the charge to NBS from EPA. Because of the variations in the approach taken in the analysis of samples from each of the four different media, these comments often pertain to only one of the four media. Additional detailed comments can be found in the answers to the template questions appearing in Appendix A.

1. "Review the sample collection and analytical protocols for organic chemicals in air, water, soils, sediments and biota to determine their appropriateness for identifying and measuring the substances of interest to EPA at Love Canal."

The approach of identifying a list of targeted substances and requiring a search for the 20 most abundant non-targeted substances

was sound. Targeted substances were chosen from appropriate lists and from the results of earlier Love Canal investigations. The criteria established for the selection of targeted substances were reasonable. However, EPA has not explicitly related included or excluded substances to specific criteria. The substances sought were representative of broad classes of organic chemicals.

Chemical identification and quantitation by gas chromatography-mass spectrometry (GC-MS) was appropriate to the general goals and objectives of the study and represented the best overall technique for monitoring organic chemicals in environmental samples. NBS is not aware of any fundamentally different analytical techniques for the identification and quantitation of organic compounds which would have been more suitable for the study as planned. For some individual substances, alternative analytical methods might have been more suitable (for example, the use of a collection medium other than TENAX for benzene and toluene--a problem recognized in EPA's draft report).

The sample collection and analytical protocols were generally complete for the air and water monitoring efforts and have been, to varying degrees, widely used in environmental monitoring for several years. The protocols for soil and sediments and for biota were modifications of the water methods and were to be evaluated during the course of the study. (The biota monitoring effort was experimental in nature and the data resulting from this effort were not used by EPA to draw conclusions from its study.) Some ambiguity existed in the details of the protocols which could have affected the quality of the analytical data.

The monitoring program design did not anticipate the possibility that a significant fraction of the samples might not contain any detectable levels of analytes. Consequently, the significance of the limits of detection and quantitation and how these limits might affect the conclusions drawn from the study were not adequately considered.

2. "Review the adequacy of the quality assurance/quality control protocols for all of the media in Task 1 to assure the validity of substance identification and analytical measurements."

The number, nature, and frequency of analysis of quality assurance samples specified by EPA should have been adequate to maintain quality control.

The quality assurance plans for the four media (air, water, soil and sediments, and biota) were similar in scope but varied in detail. The quality assurance plans for the participating laboratories were, in general, complete and followed the outline given in the overall Quality Assurance Plan. These plans did, however, vary in detail from one laboratory to another. The variations in detail could lead to

difficulties in comparing data obtained from different laboratories and different media.

The Quality Assurance Plan encouraged but did not require the use of control charts. Uniform reporting of corrective actions taken when quality control was lost was not required. It will be difficult at best to evaluate further any data reported by a laboratory without an accompanying quality control chart (or equivalent data).

The Quality Assurance Plan did not address how the quality assurance data would be used to qualify the environmental data, that is, to assign uncertainty limits to each measured value, including "none detected."

Estimates of the limits of detection are critical to the conclusions which can be drawn when the majority of samples contain no measurable concentration of analytes. The Quality Assurance Plan did not anticipate such a possibility.

3. "Review the results of the quality assurance program as revealed in documents provided to NBS describing the performance of the analytical program."

The quantitation limits realized by the water monitoring laboratories for individual analytes varied by more than a factor of ten. Few laboratories reported quantitation limits as good as those reported by the EPA laboratory responsible for the water monitoring effort. Comparisons of quantitation limits with data from other studies (where available) indicated the laboratories were not uniformly performing at the state of the art.

The limits of quantitation to be achieved by the air monitoring laboratories were set at concentration levels comparable to levels observed for some U.S. cities.

As demonstrated by the audit of the GC-MS computer records, the water and the soil and sediments monitoring laboratories did not generally identify non-targeted substances. This aspect of the performance of the air monitoring laboratories was not evaluated by EPA.

In isolated instances, contamination of the samples during sample collection or analysis may have obscured possible trends and patterns in the environmental data.

The transcription of the data and the verification of the accuracy of the transcription and transmittal of the data into the final data base were adequately demonstrated.

The documentation provided by EPA indicated that the quality control limits set in the Quality Assurance Plan widened during the study.

The degree of change was greater for the water program than for the air program. The reasons for the observed changes have not been explained in the documents provided by EPA.

The estimation of the precision and accuracy of the numerical values of concentrations of targeted substances has not been adequately documented. Only a portion of the available quality assurance and environmental data has been used in arriving at these estimates. The estimates are generally unconfirmed and estimates for some of the analytes have not been given.

The estimation of the limits of detection and quantitation are also not adequately documented. Because the conclusions of the study rest on comparisons of the frequency of compound detection, the values obtained for the limits of detection of individual substances can influence significantly such conclusions.

The rejection of data has neither been clearly described nor adequately justified.

4. "Review as necessary any of the reports of the on-site laboratory audits carried out by EPA."

The reports of on-site laboratory audits reviewed by NBS described audits conducted by EPA and by the prime contractor during the early stages of the Love Canal Monitoring Study. These reports indicate that all laboratories were having some difficulties and some laboratories were having great difficulty at the start of the study. In particular, some of the laboratories were experiencing difficulties in adjusting to the use of capillary columns.

The prime contractor maintained telephone contact with participating laboratories during the course of the study, but there is no evidence of further monthly on-site visits as prescribed by the Quality Assurance Plan. Such on-site audits would have confirmed whether or not initial difficulties had been overcome, whether or not laboratory contamination of samples was a problem, whether or not good laboratory practices were being followed, and whether or not analog data (e.g., chromatograms) from the GC-MS runs indicated laboratories were operating at the expected level of performance.

5. "Review the results of the EPA audit of GC-MS tapes to evaluate the quality of the analytical program."

The EPA audit of GC-MS computer records was not an evaluation of the quality of the entire analytical program, but only an evaluation of one aspect of that program, namely the interpretation of the computer

records. The audit evaluated a sample of the computer records generated in the water and in the soil and sediments monitoring programs. No audit of the air or biota monitoring programs was performed.

The conclusions of the audit have not been completely justified, e.g., the claim that missed compounds were predominantly in heavily contaminated samples is not supported.

The implications of the audit--that significant difficulties existed at the limits of detection--to the conclusions of the study are not discussed in documents provided by EPA.

Eighty samples (water, soil and sediments) were audited for the identification of non-targeted substances. In 58 of these samples, both the audit laboratory and the analytical laboratory identified no non-targeted substances. In the remaining 22 samples, the audit laboratory identified 84 non-targeted substances while the analytical laboratories found only 1. This result indicated that, for the samples audited, most laboratories did not identify non-targeted substances.

6. "Review any of the monitoring data as necessary to help in the overall review of the program."

Precision, accuracy, limits of detection, and limits of quantitation have not been incorporated into the validated data base. That is, the measured values, including "none detected," in the validated data base are not accompanied by estimates of their uncertainty.

The information given is not sufficient to assist others in the interpretation of the Love Canal data. Information on laboratory-to-laboratory variability is incomplete. In particular, recovery factors have not been given nor confirmed for most analytes in water and in soil and sediments samples.

In the presentation of information, the distinction between plans and attained performance is not always clear. Examples selected to support the conclusions of the study have not always been confirmed as being truly typical of the data in general.

Conclusions of no discernible patterns or differences or of no significant contamination are bounded by the limits of detection and quantitation. In the presentation of its conclusions, EPA has not addressed explicitly how those conclusions are influenced by the limitations in its analytical data. Unless measured values, including "none detected," are accompanied by estimates of uncertainty, they are incomplete and of limited usefulness for further interpretation and for drawing conclusions.

7. "Based upon written material provided by EPA, prepare a report summarizing the reviews conducted by NBS in Tasks 1 to 6, paying particular attention to conclusions drawn by EPA concerning the precision and accuracy of organic analytical data from the Love Canal Monitoring Study."

The foregoing discussion, along with additional, detailed comments appearing in Appendix A, constitute the report summarizing the reviews conducted by NBS in Tasks 1 to 6.

APPENDIX A

RESPONSES TO THE TEMPLATE QUESTIONS

Template questions were designed by NBS to serve as a guide for its review, to give structure to the review, and to ensure that all appropriate aspects of the analysis for organic chemicals in the Love Canal Monitoring Study were considered. NBS has not always answered each subquestion but has used each group of questions as guidance for its review. All questions are included for completeness.

I. Goals and Objectives for the Acquisition of Organic Analytical Data from the Love Canal Monitoring Study.

Scope: To evaluate the consistency of the goals and objectives for the acquisition of organic analytical data with existing state-of-the-art methodology.

A. What were the goals and objectives of the Organic Analysis Project? Has EPA addressed in the written documentation whether or not these goals were met?

The goals and objectives of the Love Canal Monitoring Study were presented on Page 1 of the draft report reviewed by NBS. As stated therein, these were:

1. Determine the current extent and degree of chemical contamination in the area defined by the emergency declaration order.
2. Assess the near-term and long-term implications of groundwater contamination in the general vicinity of Love Canal.
3. Provide an assessment (from an environmental contamination perspective) of the habitability of residences included in the emergency declaration order.

These goals were defined in somewhat more detail on Pages 53 and 54 of the draft report as follows:

1. To characterize in each environmental medium the incremental extent and degree of chemical contamination in the Declaration Area directly attributable to Love Canal.
2. To determine potential temporal variability in contamination and infer the causal mechanisms (for example, changes in climate) influencing the observed contamination patterns.
3. To determine if swales, sewer lines, and other geological features (for example, sandy soil deposits in the form of sand lenses) had a significant effect on the migration of toxic substances from the canal.
4. To determine the presence and direction of ground-water flow in the area, and evaluate the effectiveness of the remedial construction performed at Love Canal.
5. To investigate the use of locally available biological systems as potential indicators of toxic substances present in the environment.
6. To obtain integrated multimedia measurements of environmental contamination.
7. To provide from an environmental perspective an assessment of the relative habitability of Declaration Area residences and the short-term and long-term implications of observed environmental contamination.

In the section on implementation beginning on Page 54, immediately following the statement of the objectives of the study, it is stated:

"The EPA studies were initiated by first identifying the data requirements of the overall objectives and then designing data collection mechanisms appropriate for such activities."

These identified data requirements have neither been described further nor have they been presented in the report beyond the non-specific statements on Page 61:

"The common objective of these plans was to collect and analyze a statistically adequate number of samples to characterize accurately Declaration Area contamination caused by Love Canal, and to minimize the effects and uncertainties associated with the constrained sampling period."

and Page 62:

"...a primary goal of qualitative accuracy for organic analyses (that is, correct identification of detected substances) was established."

These statements do not address in quantitative terms the data requirements of the goals and objectives of the study. For example, the data requirements could have been made more quantitative by defining the concentrations of targeted compounds which had to be detected and/or quantified in order to compare levels of contamination in the Declaration Area with levels known to constitute an environmental hazard and/or with levels currently found in U.S. cities.

On Page 70 of the draft report, statements concerning the expected performance of analytical methodology are made:

"Precisions better than 50 percent RSD [relative standard deviation] were expected in water and air; precisions better than 100 percent were expected in other media."

"Furthermore, it is also known that in water, the minimum method quantitation limits expected for the methods used are in the range of 1 to 10 micrograms per liter (parts per billion).... Quantitation limits below these values were neither required nor expected of the analytical subcontractors, except as noted previously for 2,3,7,8-TCDD and certain pesticides."

With the exception of the 2,3,7,8-TCDD, these were expectations rather than data requirements. Without more quantitative specificity, it is difficult to see how EPA could design a monitoring program to meet its requirements.

In the presentation of its conclusions, EPA does not address explicitly how limitations in the analytical data affected the conclusions. Whether or not this is a direct consequence of the absence of quantitative precision and accuracy goals cannot be determined. Nevertheless, those who wish to use the Love Canal data or wish to interpret the conclusions of the Love Canal Monitoring Study must read and understand the entire report in detail, including the Appendices, to gain even a qualitative sense of the limitations of the data gathered in the study.

The "primary goal of qualitative accuracy" is partially addressed in the GC-MS audit performed by EPA and is discussed in more detail in Section VII of this Appendix.

- B. Would the approach of selecting a list of targeted compounds in any way hinder EPA from observing, identifying, or quantifying significant quantities of other compounds? Should EPA review all of the GC-MS tapes to determine if significant chemicals might have been missed?

The approach of selecting a list of targeted compounds should not have hindered EPA from observing, identifying, or quantifying other compounds which are amenable to GC-MS. Furthermore, this approach is appropriate to the study and the list is reasonable. By restricting the analytical

methodology mainly to GC-MS, those compounds which are either too polar or too nonvolatile to analyze without derivatization would not be detected. To derive the list of targeted compounds, EPA selected from a list of chemicals which were known to have been dumped in the Love Canal, chemicals found in previous studies of the Love Canal, and certain chemicals generally recognized as toxic, associated with industrial production, or observed in other studies as environmental contaminants--the so-called "priority pollutants". EPA should state in its report which compounds were selected from which lists. The list of targeted compounds was not all-inclusive. For example, a previous study of the Love Canal area (J. Barkley, et al., Biomed. Mass Spectrom. 7(4), 139-146 (1980)) cites estimated levels of 1,1,1-trichloroethane and trichloroethylene in air inside homes in the Love Canal area, but these compounds were not in the list of targeted air compounds in Table I-2, Page I-4 of the draft report.

All laboratories were instructed to identify the 20 most abundant substances beyond those appearing on the target list. The audit of the GC-MS computer records (discussed in Section VII below) indicated that the requirement for identifying non-targeted compounds was generally not met. Further review of the GC-MS computer records for non-targeted compounds missed in the initial analyses would be useful only if it were to become necessary to evaluate the significance of these compounds.

In the description of the development of the list of targeted compounds, it was noted:

"...the intentional inclusion of specific substances on the target list that were known to serve as effective and efficient tracers of subsurface migration of leachate was designed to permit a comprehensive determination and assessment of migration patterns from the canal source."

Nowhere in the report are these compounds identified or justified as efficient tracers, nor were any results presented which give the reader of the report an indication of whether or not these efficient tracers migrated through the subsurface area in the Love Canal region.

- C. Were the accuracy and precision goals for the organic data clearly established at the outset and have the criteria for these goals been clearly explained?

Section 3.3.2, Pages 68 to 70, of the draft report contains a description of the precision and accuracy goals for the study. This section deals primarily with the accuracy of compound identification and provides some references to the precision which might be expected for quantitative analyses such as those conducted by EPA in its Love Canal study. Precisions of 2 to 13 percent relative standard deviation (RSD) for water analyses, 50 percent for air analyses and 100 percent for other environmental media were

quoted in the draft report as generally being acceptable. These estimates of precision did not serve as limits to be used in a quality control program, but as guidelines for what could be expected from a study such as this. In its analyses of the data acquired from the Love Canal samples, EPA accepted, in some cases, data which had uncertainties as high as one or two orders of magnitude (cf. Table III-5, Page III-28 of the draft report).

Criteria (reviewed in Sections II and III below) were established for the acceptance or rejection of data and were based on laboratory analyses of quality assurance samples. However, accuracy and precision goals for the organic data were not established at the outset. The draft report includes statements of precision and accuracy which varied from one procedure to another and from one laboratory to another.

- D. If inadequacies in the precision and accuracy goals are identified in answering the above questions, will these inadequacies affect our judgment of the quality of the organic analytical data?

The establishment of precision and accuracy goals, the training of laboratory personnel to meet those goals, and the use of quality control charts based on those predetermined goals would have improved data quality.

Without precision and accuracy goals which have been formulated on the basis of the overall goals of the study, it is difficult to determine if these overall goals were actually met. Moreover, the lack of clarity in relating the precision and accuracy estimates of the acquired data to the overall conclusions of the report will make decisions based on those conclusions difficult to justify. This lack of clarity is illustrated by the following excerpt from the discussion of air contamination on Page 183 of the draft report:

"Even though maximum concentration levels are often of considerable interest to individuals, because in some way they may be thought to represent 'worst case' estimates of environmental contamination, problems of interpretation exist. The reason for this is because both the occurrence and reliability of the obtained maximum values may be plagued by measurement problems. To illustrate this point, it is often the case that maximum concentrations are reported by only one analytical laboratory and on one particular date, whereas other analytical laboratories do not report concentration levels anywhere near such maxima (and in some cases do not even report concentration levels above the minimum detection value)."

Statements such as these allude, in a general way, to the lack of precision and accuracy in the data but are not supported by quantitative information in the draft report. They give little guidance to the user of the data on how the lack of precision and accuracy affects conclusions that might be drawn.

II. Monitoring Protocols

Scope: To evaluate the consistency of the monitoring protocols with the goals and objectives of the Organic Analysis Project and with existing state-of-the-art methodology.

- A. Have the sources of the protocols used by EPA been identified? Are these protocols well documented? Had they been evaluated and did EPA consider these evaluations in their selection? Are there additional references EPA should have considered in the selection of these protocols?

The analytical methodology used in the study is described in the document "Quality Assurance Plan: LC-1-619-206, Appendix B" by the prime contractor for the EPA study, the GCA Corporation.

Those methods which were used in the water analyses, Methods 608, 624, and 625, had been, prior to the initiation of the study, published in the Federal Register. These methods were in the process of being validated by interlaboratory tests, and such validation may now be complete. To the best of our knowledge, such validation was not available at the outset of the Love Canal Monitoring Study and hence could not be used as a guide for quality control. When Methods 608, 624, and 625 were published in the Federal Register in December 1979, EPA asked for and received comments on the methods. These comments have been considered by EPA, were found in some cases to be germane, and if incorporated would have led to improvement in the clarity of presentation or in the actual performance of the methods. These modifications were not incorporated into the methods used in the Love Canal Monitoring Study. Nevertheless, Methods 608, 624, and 625 have been widely used and in our opinion represent a reasonable choice for the study.

The methods used for the air analyses are well referenced in the draft report and are described in detail in the aforementioned Appendix B of the Love Canal Quality Assurance Plan. To our knowledge these methods have not been formally validated.

The sample preparation steps in the methods used for soil, sediments and biota samples were experimental in nature and had to be developed as part of the Love Canal Monitoring Study. They were, consequently, unevaluated beyond the evaluation appearing in the draft report. A number of the protocols which, taken together, comprised the methods for the soil, sediments and biota were still optional at the time Appendix B of the Quality Assurance Plan was released. Although the methodology was fixed by the end of the study, it is unclear whether all the data acquired for the soil, sediments and biota were obtained using the methodology described in the draft report.

There are no fundamentally different analytical techniques for the detection and quantitation of organic compounds which would have been more suitable for the study as planned.

- B. Does EPA discuss the precision and accuracy to be expected from the chosen protocols? Does EPA document the minimum detectable limits for their targeted compounds? Are the precision, accuracy and minimum detectable limits claimed by EPA reasonable and consistent with our knowledge of or experience with the methodology? Are these claims supported by reference to available literature?

Since none of the methods used in the study had been subjected to a formal interlaboratory validation prior to the study, expectations concerning the precision, accuracy and minimum detection limits had to be drawn from the reported experience of analysts who had used the various methods.

For air methods, EPA established limits of quantitation for targeted compounds; limits of detection were to be determined by each laboratory. For water methods, the limits of quantitation for targeted compounds were to be determined by each laboratory; expected limits of detection were reported in the Federal Register description of the methods.

For many of the protocols, expectations of precision, accuracy, and detection limits were unavailable and were to be determined during the course of the study from the quality assurance data. The protocols did not, in all cases, explain how such determinations were to be made. Moreover, such guidance as was given varied from one environmental medium to another. For example, precision was to be determined for air monitoring methods from data on duplicate measurements and sample splits and the accuracy from data on spiked TENAX tubes and polyurethane foam plugs, while for total organic carbon determinations in water, the methods called for the analysis of blanks and replicates but had no requirement for the determination of accuracy.

Not all claims on expected precision, accuracy, and detection limits were supported by appropriate references. The requirement that they be determined by the individual laboratories during the course of the study is correct and always necessary.

- C. Can we identify targeted or non-targeted compounds which the monitoring protocols would be likely to miss? Can we recommend ways in which EPA could justify the completeness of its identification of compounds which it has not presented in the written documentation supplied to NBS?

In general, the protocols were capable of detecting all targeted compounds. Because of the composition of TENAX, and the allowed lapse time between collection and analysis of samples, the use of TENAX for the analysis of benzene and toluene in air is inappropriate at the levels encountered. This problem has been recognized by EPA and is commented upon in its draft report.

The protocols are likely to miss certain non-targeted compounds. In particular, compounds which are too polar or not sufficiently volatile will not be detectable using GC-MS.

D. Are the protocols as written complete? Do they omit steps or precautions that would be expected to affect the identification or quantification of certain organic compounds? Were all of the following steps clearly described in the protocols, were they complete?

1. Sample Collection Techniques
2. Sample Storage and Transportation
3. Chemical and Physical Manipulation of the Samples Prior to Analysis
4. Calibration
5. Analysis
6. Records

The protocols were sufficiently complete in the sense that they could be followed by competent analytical chemists with experience in organic analysis. The protocols for the sample collection, for storage and transportation, and for record keeping were satisfactory. Some ambiguities in the remaining steps could have resulted in actual differences in how the protocols were executed. Such differences could introduce difficulties when comparing data from the various laboratories.

Instances of incompleteness or ambiguity in the protocols are listed below.

- (1) The analytical procedure for volatile organics on TENAX allows for the use of an internal standard, dependent on the availability of the apparatus needed to perform spiking (Quality Assurance Plan, Appendix B, Section 1.2.2C). This apparatus is not defined in the procedure and the addition of an internal standard is optional. Comparability of data between laboratories requires comparable methods of internal standard addition.
- (2) Mass spectrometric confirmation of gas chromatography-electron capture (GC-EC) and high performance liquid chromatography (HPLC) measurements for pesticides collected on polyurethane foam plugs are to be conducted on composited or selected samples. The choice of these samples is not

specified. Furthermore, Subsection F, Section 1.3.1, Appendix B of the Quality Assurance Plan of this analytical procedure directs the analyst to "confirm results as required by combined GC-MS" but does not specify what constitutes "as required."

- (3) The analyst is given the option of choosing from four methods, involving different extraction schemes and packed or capillary columns for the analysis of semi-volatile organic compounds in water.
- (4) The analytical procedure for the determination of total organic carbon in water samples requires a chemical preservation step to be performed in the laboratory within two hours of collection. The QA procedure submitted by JRB Associates specifies that samples to be analyzed for total organic carbon and total organic halogen content are to be transferred to the GCA sample bank within four hours of collection.
- (5) The analytical method for the determination of pesticides in sediments calls for the use of a procedure entitled "Sample Preparation and Analysis of Bottom Sediments" with the exception that part 5 of the method has been modified and part 4 of the method is to be disregarded. There is no statement of what is to be done in place of part 4. (Part 4 is the section dealing with sample preparation and extraction procedures.) One could assume that Method 608 is the replacement, but this is not specified. In this same bottom sediments procedure, the gas chromatographic step calls for proceeding as described in Section 11.A. There is a Section 11 of Method 608 which deals with gas chromatography but there is no subsection 11.A.

E. Do the protocols adequately address the prevention of sample contamination from impure reagents, the sampling process, contaminated sample containers, etc.?

With few exceptions, the protocols dealt with the problems of analytical contamination in a reasonable manner. These exceptions included the problems of benzene and toluene contamination of TENAX mentioned in II.C above and problems with vagueness or inconsistency in instructions on how to correct for common sources of analytical contamination. For example, the quality assurance procedure for the analysis of semi-volatile compounds in soil and sediment required that the method blank not show any signal corresponding to compounds on the list of targeted compounds, but if common laboratory contaminants such as phthalate esters (which were on the targeted list) are found, the reported values for the corresponding compounds in the environmental samples must be reduced by the amount found in the blank. This instruction is vague in that levels of contamination for which method blank corrections are valid are not given.

- F. In the documentation provided by EPA, is the intended use and purpose of the data from the control samples described? Is this intended use reasonable? Could it have been expected to achieve its intended purpose?

The control samples, that is, samples from the Control Area, were designed for comparison of levels of contamination in the Declaration Area with levels outside that area. (As such, they were not intended as analytical controls and should not be interpreted as such. They are, within the context of a review of the analytical program, to be treated as environmental samples, indistinguishable from any other environmental samples.) Their use and purpose are described and the intended use is reasonable. Determination of whether or not they were sufficient in number and kind to have achieved their intended purpose is beyond the charge from EPA to NBS.

- G. Are there any means, not addressed above but available to EPA at the beginning of the study, by which EPA could have improved upon the selection, description or justification for use of the selected monitoring protocols?

Improvements in the methodology have been addressed in the sections above.

III. Quality Assurance Protocols

Scope: To evaluate the consistency of the quality assurance protocols with the accuracy and precision objectives of the study and to evaluate the adequacy of these protocols to maintain quality control over the reported data.

- A. Was the quality assurance plan sufficient to maintain quality control? If not, what were the major flaws in the plan? Were there any necessary elements missing?

A major feature of the Love Canal Monitoring Study was the quality assurance program for the study. The prime contractor, GCA, prepared an extensive quality assurance plan, and all but one of the participating subcontractors prepared quality assurance plans in response to the GCA plan. These plans contained a large variety of quality assurance measures including document and report control, laboratory control standards, replicate analyses, internal standards, calibration checks, and surrogate standard additions.

Quality assurance must be recognized as consisting of two factors: quality control and quality assessment. The quality control aspects of the plans were generally adequate, especially those portions which dealt with the collection and handling of samples and maintenance of records, including chain of custody. The Quality Assurance Plan was deficient in the manner in which it addressed quality assessment. The interrelation of individual items and the manner in which quality assurance data was to be analyzed and used were not clearly described.

Protocols varied between media. Examples of this variability from Section 7 of the Quality Assurance Plan will be described in the following paragraphs.

- (1) For air samples collected on TENAX cartridges, the use of an internal standard was optional and this standard when used was to be added to the TENAX cartridge. Internal standards should have been mandatory. For air samples collected on polyurethane foam plugs, an internal standard was mandatory, but was added to the sample extract. Moreover, the procedure stated "if a suitable compound can be determined a single internal standard will be added to each sample extract." The standard should have been specified and should have been added as early as possible in the analytical procedure. For the determination of dioxin (TCDD) in air samples, chlorine-37 labeled TCDD was added to each sample as an internal standard. This was appropriate.
- (2) For those air samples collected on polyurethane foam plugs, a three point calibration curve of all compounds was required each day; a single-concentration, mixed standard was required after every fifth

sample; and one spiked foam plug was to be analyzed per analysis batch per day. For those air samples collected on TENAX, only a single calibration check was required per eight hours of analysis. No discussion is presented describing why the two protocols were so different.

- (3) For the determination of semi-volatile organic compounds in water, deuterated internal standards were required (but the draft report contains no mention of their use). For the determination of volatile organic compounds in water, internal standards (non-deuterated) were optional. For the determination of pesticides in water, the analyst is referred to Section 7 of Method 608 which recommends the use of fortified samples. The use of internal standards should have been required.
- (4) For the determination of volatile and semi-volatile organic compounds in water, surrogate compound additions were required. Recoveries of surrogate compounds were to fall within specified ranges. (The ranges described in the draft report were substantially different from the ranges in the Quality Assurance Plan.) For pesticides, the use of fortified samples was recommended (by reference to Method 608).
- (5) For the determination of pesticides, volatile organic compounds, and semi-volatile organic compounds in water, laboratory control standards were required. Control limits were established for the recoveries of the laboratory control standards. For pesticides, the control limits were specified as " ± 2 standard deviations." For volatile and semi-volatile organic compounds, the control limits were specified as ranges of "percent of true value" for individual compounds.
- (6) For the analysis of pesticides in soils and house dust, a recovery check on a spiked sample using a list of specified pesticides was required. The same recovery check was not part of the sediment procedure and should have been.
- (7) For the analysis of total organic carbon and total organic halogen content in water, specifications were presented for method blanks and replicate analyses, but none for a laboratory control standard, an internal standard or a surrogate standard; hence, there was no accuracy check.

In addition, control limits associated with many of the quality assurance procedures were to be determined during the course of the study. These limits, if they were determined, have not been reported. Performance as determined from calibration check samples and performance evaluation samples was to be evaluated as the study progressed. This evaluation process was not described in the Quality Assurance Plan.

The above remarks indicate the quality assurance plans were quite extensive and detailed, but were lacking in consistency from one medium to the next and lacking in detail on how the quality assessment was to be performed.

- B. Were criteria established for the rejection or acceptance of data? Were these criteria reasonable, and if not, how could they have been improved?

Two levels of review for rejection or acceptance of data were included in the Love Canal Monitoring Study. The Quality Assurance Plan called for contract laboratories to monitor their own performance and to invalidate data according to certain criteria. Laboratory performance was to be reviewed by the prime contractor and by EPA. Criteria for acceptance or rejection by the prime contractor or by EPA of data already validated by the contract laboratories were not part of the Quality Assurance Plan, but were described in the draft report. Furthermore, these criteria were not consistent with those in the Quality Assurance Plan, as will be described in the following paragraph. In contrast to the statement on page 64 of the draft report, the Quality Assurance Plan did not require the contract laboratories to maintain and submit quality control charts.

The criteria used by EPA for acceptance or rejection of data differed from the criteria specified for the contract laboratories in the Quality Assurance Plan. For example, Table 7.2.1 of the Quality Assurance Plan specifies control limits for the recovery of surrogate compounds in water of 81 to 100 percent for fluorobenzene, 88 to 118 percent for p-bromofluorobenzene, 18 to 58 percent for 2-fluorophenol, 33 to 88 percent for 1-fluoronaphthalene, and 34 to 98 percent for 4,4'-dibromooctafluorobiphenyl. Table III-5, Page III-28 of the draft report indicates there were no upper control limits for the recovery of these five surrogates and that EPA invalidated all analyses for which more than one of the surrogates was lower than the lower control limits of 68 percent, 60 percent, 1 percent, 2.8 percent, and 8.3 percent respectively for the five compounds mentioned above.

The EPA criteria for validation of data were less stringent, in practice, than the criteria in the Quality Assurance Plan. Whether this indicates the plans were unrealistic or the implementation of the Quality Assurance Plan was inadequate cannot be determined.

- C. Were the quality assurance plans of the individual contractors uniform and consistent with each other and with the overall Quality Assurance Plan?

The quality assurance plans of the individual contractors followed an essentially consistent outline which was appropriate to the study. The individual plans, however, differed in detail.

Not all quality assurance plans were available or complete when NBS began its review. In particular, to our knowledge, no plan was filed by Research

Triangle Institute (RTI) which was one of the three QA contractors and which had responsibility for the spiking of the TENAX cartridges used in the QA program for the analyses of air samples. The QA plans from TRW, one of the laboratories performing analyses for organic compounds in water, and Wright State University, the only laboratory performing dioxin analyses, were received late by NBS and it is not clear whether these plans had received approval from EPA prior to the initiation of the Love Canal Monitoring Study. The plans received from CompuChem/Mead and Accurex Corp. contained notations that additional information had been requested. This information was received by NBS well after completion of the chemical analyses. It is not clear whether these additions were included in their QA plans or were descriptions of practices which had been followed. The plans from PJB and Advanced Environmental Systems were lacking in a number of elements required by the overall QA plan of the prime contractor, but no indication was given whether revised plans or additional information had been requested. The QA plan for Southwest Research Institute provided no information on the analytical procedure or the quality control procedure for the preparation of the polyurethane foam plugs which were used in the air analyses. The plan from Battelle Columbus Laboratories lacked a summary of analytical methods, equipment maintenance, and trouble-shooting procedures. Finally, none of the QA plans addressed the identification of the 20 most abundant, non-targeted compounds.

The quality assurance plans should always be signed and dated by the submitting laboratory and the approving authority before work begins. This was not done consistently in the Love Canal Monitoring Study.

- D. Was the number, nature and frequency of quality control samples or performance audit samples appropriate to achieve monitoring goals, to take corrective action, and/or to maintain quality control?

The number, nature, and frequency of quality control samples as required by the Quality Assurance Plan should have been adequate to maintain quality control. Evidence exists, however, that quality control was not maintained in some cases. The Quality Assurance Plan did not anticipate the large number of samples which would be found to contain no levels of contamination above the limits of detection. When a large number of samples are expected to contain concentrations below the detection limit, sample splits and duplicate samples are of limited usefulness. These difficulties will be discussed further in Section V dealing with implementation.

E. Did the plan adequately and clearly prescribe corrective action?

Instructions from EPA and the prime contractor to the contract laboratories for corrective action (CA) were clearly and adequately described in the overall Quality Assurance Plan with two exceptions: (1) The overall plan did not require contract laboratories to maintain and submit quality control charts. (2) There existed ambiguity in the description of the mechanism for reporting corrective action to the prime contractor. Figure 11-1, Page 11-3 of the Quality Assurance Plan, is an example of a form which was to be completed and submitted describing corrective action taken whenever loss of quality control was identified. On Page 11-4, however, it was stated "subcontractors are not required to follow this system exactly or to use the pictured CA form." For such a large and complex study conducted in such a short period of time, a uniform system should have been required. On the basis of the written, corrective-action procedures appearing in the individual subcontractor QA plans, it is apparent that corrective-action procedures were not consistently adequate. For example, IIT stated corrective action (unspecified) would be implemented as required but did not state that it would report the need for corrective action to GCA. PEDCO also gave only a vague statement of the nature of corrective-action procedures and the documentation of corrective action.

IV. Performance of the Monitoring Program

Scope: To evaluate, from the information provided by EPA, whether or not the implementation of the monitoring program met the goals and objectives of the Organic Analysis Project and were consistent with proper implementation of the monitoring protocols and with commonly accepted good laboratory practices.

- A. What evidence does EPA present to support claims of proper implementation of the monitoring protocols? Is the evidence presented that the contract laboratories were capable of performing their assigned tasks convincing?

Based on the material reviewed, the sample collection, storage and transportation, document control/chain of custody procedures, and data management were generally implemented according to plan. Field and quality assurance samples were distributed according to plan. An acceptable fraction of the samples was analyzed. Some difficulties with the analysis of the samples were encountered and these difficulties are discussed in the remainder of this Section and in Section V below.

- B. Is there any reason to believe that significant concentrations of targeted or non-targeted compounds were missed in the analyses of the samples? What additional information should EPA provide to justify claims that compounds were correctly identified and that significant concentrations of targeted or non-targeted compounds were not missed?

The GC-MS audit of computer records indicated that most non-targeted compounds were probably missed. Pages VI-12 to 13 of the draft report state:

"In most of the 22 samples [of the 80 samples audited] containing non-target compounds, the audit laboratory reported finding 1 or 2 compounds while the analytical laboratory reported none. There were 5 samples audited where 6 to 20 compounds were reported by the audit laboratory but none were reported by the analytical laboratory...."

The audit also indicated inconsistency between laboratories in the determination of the presence of targeted compounds when the concentration of those compounds was near the detection limit.

Pages III-14 to III-16 contain a discussion of the limits of detection and quantitation for organic compounds in water. Table III-1, Page III-15, contains a summary of the limits of quantitation, and the accompanying discussion states:

"There was considerable variance among the analytical laboratories in method quantitation limits for a given analyte, and the data suggest that some laboratories were not operating consistently at the state-of-the-art possible with the methods. This is neither unusual nor unexpected."

Comparison of the numbers in Table III-1 with:

- (a) Table II-1, Page II-5 which contains the range of median values of compounds found in drinking and surface waters in various industrial locations,
- (b) Table II-4, Page II-8 which contains values of volatile organic compounds found in drinking water in a number of cities, and
- (c) Table II-9, Page II-34 which contains values of selected volatile organic compounds found in national surveys of water supplies,

indicates that the limits of quantitation for volatile organics, determined by three of the five laboratories performing water analyses in the Love Canal Monitoring Study, were an order of magnitude higher than values reported by laboratories participating in the studies quoted in Appendix II of the EPA draft report. Also, for the volatile organics, the limits of quantitation for the five contract laboratories were 1, 2, 4, 8 and 9 times higher than those of EPA's own laboratory in Cincinnati. For the semivolatile organics, little data exist against which to compare the results of this study, but the limits of quantitation for the four contract laboratories were 1, 8, 9, and 14 times higher than those of the EPA laboratory. In its draft report, EPA recognizes the variability in limits of quantitation reported by the contract laboratories. This variability will make the Love Canal monitoring data difficult to interpret.

The limits of quantitation set for the air monitoring laboratories appear to be adequate for quantifying levels of compounds which have been reported for some U.S. cities.

C. Is there any reason to believe that significant contamination of the samples from impure reagents, the sampling process, or contaminated tools, or containers may have occurred? Does EPA present evidence that this has not occurred? Is it convincing and if not, what additional documentation is needed?

EPA has presented evidence that artifactual contamination, that is, contamination of the samples from impure reagents, the sampling process, or from contaminated tools or containers, was a problem for certain samples and for identified analytes.

- (1) For those air samples collected on TENAX, significant blank problems associated with benzene, toluene, and 1,1,2,2-tetrachloroethylene were encountered. In Table V-4, Page V-13 of the draft report, EPA indicates

the frequency and concentrations of these compounds found on blank TENAX cartridges. The accompanying text states:

"To be relatively certain that an obtained single value was not due to blank contamination, the field concentration should be greater than three standard deviations above the mean values reported in Table V-4 for these three compounds."

From the data in Table V-4, "three standard deviations above the mean values" were 7, 29, and 22 micrograms/cubic meter for benzene, toluene and 1,1,2,2-tetrachloroethylene respectively. According to the draft report analytical values obtained for these compounds below these levels would be suspect. (Averages of several analyses would have somewhat lower limits depending on the number of samples included in the analyses.) In Section 4.2.6, Pages 167 to 195 of the draft report, EPA describes the results of the air monitoring program. The only significant differences between the Declaration, Canal and Control Areas are the concentrations of o-chlorotoluene, o-dichlorobenzene, and chlorobenzene. Yet, for the remainder of the discussion, only benzene, toluene and 1,1,2,2-tetrachloroethylene are discussed. We question the appropriateness of using these three compounds, for which blank problems call into question the reliability of their determination, as "typical" examples. The median concentration plots in Figures 54, 55 and 56 on Pages 188 to 190 were chosen to illustrate the lack of pattern in the distribution of these compounds, but the maximum values for these medians are below the above-mentioned limits on TENAX blanks.

- (2) On Page III-29, the contamination of bedrock B wells by use of ordinary hydrant water during the drilling process is described. This led to the exclusion of "28 ground-water, Method 624 samples" from the validated data base. Chloroform was identified as the contaminant and its source was attributed by EPA to improper purging of the wells, but no quantitative data were presented to support this claim. A total of 340 bedrock well samples were included in the validated sample set. The effect of excluding the 28 samples on the conclusions drawn by EPA is not discussed in the draft report.
- (3) On Pages III-29 to 31, the contamination of water samples by methylene chloride, bis(2-ethylhexyl) phthalate, and dibutyl phthalate is described. This contamination resulted from the presence of these chemicals in the atmosphere of the analytical laboratories and from plastic containment materials. As a result, all reports of these three analytes were removed from the validated data set. The same problem was encountered with the sediment and soil analyses.

With the exception of item (1) above, the incidence of artifactual contamination should present no problem so long as conclusions drawn concerning Love Canal are based on patterns observed for groups of chemicals and not for individual substances. The above examples of contamination were discovered because so many of the samples which were collected had no concentrations of analytes above the detection limit. For the same reason,

it is unlikely there are other cases of widespread, but as yet undetected, artifactual contamination by targeted compounds.

- D. Is there any evidence to indicate that participating laboratories were incapable of adequately following the protocols or did not adequately follow the protocols? Is there any evidence of carelessness in execution of any of the steps from sample collection to analysis?

As part of the quality assurance program, site visits were made to the participating laboratories and reports of these visits were reviewed by NBS. The reports indicated that most laboratories were having some difficulties at the outset, and a few were having great difficulties. In particular, some of the laboratories were experiencing difficulties in adjusting to the use of capillary columns. No follow-up visits were made, or if they were, reports of such follow-up visits were not provided. It is not known if appropriate corrective action was taken in all cases.

The data in Table 6 on Page 110 of the draft report are inconsistent with the number of targeted compounds which were actually sought in water, sediment and soil samples. Table 6 contains a summary of the frequency of detection of substances in the validated Love Canal samples. The table also contains the "number of substances (analytes) measured" and the number of samples which were analyzed. Dividing the number of analytes by the number of samples gives the average number of analytes per sample. These average numbers are, for the air analyses, consistent with the number of targeted analytes listed in Table I-2 on Page I-4. For the water and sediment analyses, these average numbers range from 20 to 30 for almost all of the samples analyzed, whereas Table I-1 on Pages I-1 and I-2 indicates that 135 analytes were to be sought. Similarly, the average number of analytes per sample of drinking water obtained from Table 12, Page 196, is 25 to 28. This inconsistency should be corrected.

Performance qualification of all the laboratories prior to initiation of the study, as was done for air monitoring subcontractors, would have diminished the number of problems encountered but could have delayed the initiation of the study. This has been acknowledged by EPA.

- E. Does EPA, in its report, adequately document the performance of the contract laboratories? If not, what additional documentation should EPA provide?

Appendix III of the draft report does not adequately document the performance of the water monitoring laboratories. Four types of performance checks were used. Laboratories analyzed laboratory control standards (LCS) and quality control (QC) samples daily, performance evaluation (PE) samples monthly and surrogates for every analysis. The laboratory control standards will be discussed in Section V.B. The QC and PE samples were concentrates of selected compounds in organic solvents which were added to reagent water

and analyzed. The identities of the compounds in the QC samples may or may not have been known to the performing laboratories; the concentrations were unknown. The identities and concentrations of compounds in the PE samples were unknown to the performing laboratories. The surrogates were known amounts of known compounds added to each sample. No summary of laboratory performance on QC samples is given in the draft report and should be. The performance on QC and PE samples was not used to determine precision and accuracy for the methods but should have been used, at least, to confirm the estimates of precision and accuracy.

Performance on PE samples is summarized in Table III-4 on Page III-22 of the draft report as percentages of analyses submitted by the contract laboratories which were judged "acceptable." The definition of "acceptable" is not given and should be. The percentage of all analyses of PE samples found to be unacceptable cannot be determined because the numbers of analyses corresponding to the entries in Table III-4 are not given. Thus, it is not possible to compare the performance on PE samples with the overall performance, i.e., the percentage of Love Canal data which was validated. The performance on PE samples should be consistent with the data validation process and this consistency should be documented and discussed. As Table III-4 now stands, it appears that the number of PE sample analyses judged as unacceptable (ranging from 0 to 75%) is considerably larger than the number of samples invalidated on the basis of surrogate recoveries (17 samples). This apparent inconsistency should be clarified.

The documentation of the performance of the air monitoring laboratories appearing in Appendix V of the draft report is generally acceptable though Table V-5 on Page V-20 would be more readily interpretable if it contained the ranges of the concentrations of the compounds to which the standard deviations pertained. Inconsistencies exist in the main body of the draft report in the description of the performance of the air monitoring laboratories. On Page 174, is the statement:

"The analytical accuracy...for the TENAX samples was within ± 10 percent (as indicated by analytical recovery of substances purposely introduced into samples at concentration levels known only to EPA)."

On Page 184 is the statement:

"As can be seen from the results displayed in Figures 52 and 53, variability in concentration levels were observed over time. As noted above, however, much of the variability observed in the sample results could be accounted for by the systematic differences in analytical laboratories and by measurement error."

The variability in Figures 52 and 53 ranges from 0 to $40 \mu\text{g}/\text{m}^3$, well beyond the $\pm 10\%$ "accuracy" claimed on Page 174. Moreover, there is no quantitative justification given of the statement "...could be accounted for by the systematic differences in analytical laboratories and by measurement error." These statements, as they stand, appear to be in conflict in their description of the performance of the air monitoring laboratories.

The documentation of laboratory performance would be improved if the performance of individual laboratories on all of the different classes of quality assurance samples were summarized, tabulated, and compared with criteria established for performance in the Quality Assurance Plan.

V. Performance of the Quality Assurance Program

Scope: To evaluate from the information provided by EPA whether or not the implementation of the quality assurance program was adequate to maintain quality control of the Organic Analysis Project and whether or not accuracy and precision claims, if given, have been convincingly justified.

- A. Were the quality assurance samples sufficient in kind, number and reliability to test adequately the performance of the participating laboratories? What sources of measurement error, if any, might EPA have missed or have had the most difficulty in evaluating?

In general, we believe the quality assurance samples were sufficient in kind, number and reliability to test adequately the performance of the participating laboratories. They should also have been sufficient to maintain quality control provided appropriate corrective action was taken. The large number of samples containing no analytes above the limits of quantitation limited the usefulness of replicates for evaluating the comparability of analyses performed in different laboratories (e.g., estimating interlaboratory precision).

The measurement errors most difficult to evaluate are those associated with the soil, sediment and biota samples. The quality control samples used for the monitoring of these samples were water samples which do not duplicate the matrix effects presented by soils, sediments and biota. Thus, not all of the steps in the analytical methodology were tested by the quality assurance program. This is acknowledged by EPA in its draft report.

- B. Has the rejection or acceptance of data from a participating laboratory been clearly explained and justified? Can we suggest ways in which the rejection of data might distort the representativeness of the data?

The rejection of data has not been clearly described and adequately justified.

The fractions of samples rejected for various reasons in arriving at the final set of validated samples are summarized in Table 1 on Page 11 of the draft report.

- (1) In Table 1 EPA indicates 410 samples out of 2457 water samples which were analyzed were rejected. The extensive discussion on the data validation process for water samples on Pages III-25 to 29 states 17 of the Method 624 and 625 samples were rejected. Discussions for the

other analytes are vague but indicate similarly small numbers of rejected samples. Therefore, it appears about 300 samples were rejected without a corresponding, satisfactory explanation in the draft report.

- (2) The percentage figures in the fourth and fifth columns of Table 1 do not agree with the totals listed in the left three columns. Moreover, they do not agree with the numbers of rejected samples appearing in the appendices. For example, from Table 1, a comparison of samples analyzed with samples validated for soil, sediments and biota give 27, 4, and 8 samples rejected, presumably on the basis of the validation procedure. Appendix IV, Page IV-23 reports these numbers as 9, 7, and 7 respectively.
- (3) The footnote to Table 1 should apply to the column headed "Percent Other" and not "% Rejected by QA/QC."

Beyond these observations, the following difficulties with the justifications of the rejection or acceptance of data were encountered:

- (1) Page III-24 contains a discussion of the errors which could result from holding samples to be analyzed by Method 624 beyond the maximum allowable holding time of 14 days. Experiments to determine the effects of holding time were performed using samples containing 100 micrograms/liter concentrations of selected analytes. The report should address and justify the significance of these experiments to samples containing 10 micrograms/liter, that is, concentrations near the limits of quantitation.
- (2) The discussion on data validation procedures (Pages III-25 to 33) and on estimates of data accuracy (Pages III-36 to 38) for the water monitoring effort should contain a description of the percent recoveries for the analytes studied. The reporting of data without correction for the lack of complete recovery of particular analytes has been justified on the basis that the analytical methods do not provide a procedure to correct for these losses. Recovery information is critical for proper assessment of data resulting from the Love Canal Study. If the mean recovery for a particular analyte were 50%, then, on the average, all data for that particular analyte would tend to be low by a factor of two. This could be important when comparing the distributions of two different analytes. The only indication of such recoveries is given in Table III-5 on Page III-28. These data were derived by EMSC and are probably not representative of the performance of the contract laboratories.
- (3) The description of the rejection of the 28 ground-water, Method 624 samples from bedrock B wells should be clarified. How did the Environmental Research Laboratory in Ada, Oklahoma determine these wells were not purged adequately prior to sampling?

- (4) As discussed in Section III.B above, criteria were established by EPA for validation of data by the water monitoring laboratories based on recoveries of surrogate compounds. EPA used the recoveries of the same compounds but substantially wider acceptance limits for its validation of sample data which should have been rejected (as required by the Quality Assurance Plan) by the contract laboratories. These wider limits were based on a statistical analysis of the recoveries measured by EMSC in 5% of the water, soil and sediments samples. The reasons why wider acceptance limits were used are not discussed in the draft report. A comparison between the number of samples, for which the data would have been validated had the original control limits been used, and the final number of validated samples would likely give greater insight into the quality of the water data than the tabular data presented in the draft report. As mentioned in Section III.E above, laboratory performance on PE and QC samples should be consistent with the sample validation procedure.

Similar, though not as serious, concerns exist for the air monitoring data.

- (5) Table III-6 on Page III-35 of the draft report presents relative standard deviations for replicate measurements of Method 624 and Method 625 analytes in laboratory control standards (LCS). Table 7.2.1, Pages 7.2.2 to 7.2.4 of the Quality Assurance Plan lists control limits for the analyses of laboratory control standards. If the analysis of the laboratory control standard yielded values outside the control limits, the laboratory was directed to check analytical procedures, analyze a second reference sample and, if still out of control, invalidate results. A comparison of Table III-6 with the control limits for the LCS's indicates the performance for certain of the laboratories was out of control for a significant fraction of the time. For example, the control limit for benzene (item 6 of Table 7.2.1 on Page 7.2.2) was 79 to 107 percent recovery, a range of 15% around the average of 93 percent recovery. The relative standard deviations of the recovery of benzene for two of the six laboratories included in Table III-6 were well in excess of this $\pm 15\%$ control limit. Similar statements can be made about other compounds listed in Table III-6. EPA should, in its report, explain this apparent discrepancy in the quality control program.
- (6) The quality assurance program for the air samples addressed the problem of artifactual contamination of TENAX by benzene and toluene if samples were analyzed more than 30 days after final cleaning of the TENAX. The significance of such contamination could be better assessed if experimental results were presented showing variability in background levels of these compounds as a function of time after cleaning.

Finally, EPA has not used the full range of quality assurance data available to it to justify its acceptance of data. The incomplete use of quality assurance data is discussed in other sections of this Appendix.

C. What conclusions has EPA drawn from the sample splits and the intercomparison of data from single sampling sites as to the precision of the data? Are such conclusions justified? If not, what conclusions could be drawn and/or what justification should be presented to support such conclusions?

EPA has drawn few conclusions concerning the precision of the data from the sample splits and intercomparison of data from single sampling sites. This is primarily a consequence of the small fraction of samples containing concentrations of analytes above their detection limits.

Triplicate samples were collected by the EPA contractors in the water monitoring program. Five percent of the samples taken consisted of such triplicates. Seventy-five percent of the samples analyzed with Method 625 contained no analytes above the quantitation limit. This would imply that only 25 of the triplicate samples had quantifiable concentrations of analytes present (25% of 5% of the total number of validated water samples listed in Table 1 on Page 11 of the draft report). In the draft report, EPA states that similar observations were made with the Method 624 and the Method 608 analytes. EPA concluded that this number was too small to give a reliable estimate of method precision. The actual number of triplicate samples containing quantifiable concentrations of analytes should be reported by EPA along with estimates of precision derived therefrom and these estimates compared with the estimates in Table III-6 on Page III-35 and with additional estimates made from quality control and performance evaluation samples.

Duplicate samples collected in the air monitoring program were used to estimate analytical precision. The precision was based on the standard deviation of the observed differences in reported values. This procedure has the desirable feature of taking into account all aspects of the monitoring process. The three compounds listed in Table V-5 on Page V-20 of the draft report for which a large number of duplicates were obtained were the three compounds identified in this study to be artifacts on the TENAX columns: benzene, toluene and tetrachloroethylene. The standard deviations appearing in Table V-5 for these three compounds reflect both the variability in analysis and in the actual concentrations of the three compounds as artifacts on TENAX. Therefore precision estimates deduced from Table V-5 for these compounds are not transferable to other compounds. These difficulties in interpreting the significance of data from duplicate samples have been pointed out by EPA on Page V-12 of the draft report.

No data are presented on those replicates which might give insight into the measurement process near the limit of detection, i.e., for cases where the detected compounds in at least one of the replicates are reported at trace concentrations. Comparison of replicates for which at least one of the compounds was reported as below the quantitation limit should be tested for consistency with the limits of quantitation reported by EPA.

D. What are some possible limitations of the quality assurance program?

The major difficulty which will be encountered in further analyses of the data acquired at Love Canal will be coping with laboratory-to-laboratory variability in performance. No additional limitations in the program have been found beyond those discussed in the answers to the other questions of this section. In summary, EPA has not used all of the quality assurance data it has collected in assessing the performance of the analytical laboratories, i.e., tables of precision, accuracy, and detection limits are based on limited sets of quality assurance data and have not been confirmed.

E. Were the abilities of the participating laboratories to detect trace levels of targeted and non-targeted compounds adequately tested by the quality assurance program? What evidence does EPA present to justify claims of laboratory performance? Is additional evidence necessary to support such claims and, if so, what?

The quality assurance program did not adequately address the question of minimum detection limits. Each of the participating laboratories was asked to estimate its limit of quantitation (for the water program) or detection (for the air program). These estimates have been tabulated in the draft report. The ability of the participating laboratories to detect trace concentrations (that is concentrations between the limit of detection and the limit of quantitation) was not formally evaluated as part of the Love Canal Monitoring Study. As stated in Section IV.B above, the participating laboratories did not appear to be achieving the levels of detection with Methods 624 and 625 which had been achieved in other studies. Greater attention should be paid to the estimates of the limits of detection. These estimates should be confirmed with other quality assurance data wherever possible. The methods used to estimate and confirm the limits of detection should be clearly described. This point is critical because all conclusions of the draft report are bounded by the limits of detection achieved in the study.

The ability to detect and identify non-targeted compounds was evaluated by the subsequent GC-MS audit (see Section VII below).

F. Beyond the acceptance or rejection of data, what conclusions has EPA drawn from the analysis of quality control samples, field blanks, and samples from control areas as to the precision and accuracy of the reported data, the overall performance of the participating laboratories, or the minimum detectable limits of targeted or non-targeted compounds? Are these conclusions justified? If not, what

conclusions could be drawn and/or what evidence should be presented to justify such conclusions?

EPA has used a portion of the quality assurance data to estimate precision, accuracy, and limits of detection for the acquired Love Canal data. Estimates of these quantities were supplied by EPA or by the contract laboratories with little documentation as to how the estimates were made. The conclusions of the study may be sensitive to patterns and comparisons near the limits of detection, and therefore the determination of these limits must be described with greater clarity and the limits confirmed. Precision estimates were based on different types of data for air, water, soil and sediments, and biota samples. For the air samples, the precision estimates were based on comparisons of duplicate samples (see Section V.C above). For soil and sediments and for water samples, the estimates of precision were based on the analysis of laboratory control standards (LCS). Not all targeted analytes were included in the laboratory control standards. Table III-6 on Page III-35 lists the relative standard deviations (RSD) for the recovery of compounds in the LCS's. Some entries are missing, suggesting lack of adherence to quality assurance protocols. Table III-6 would be improved if it included the number of LCS's for each compound for which relative standard deviations were calculated. As noted above, EPA should also use the data from quality control and performance evaluation samples to confirm the claims of precision and accuracy appearing in the draft report. These same comments apply to the precision estimates for the analyses of soil and sediments.

The accuracy of air methods was estimated from extensive studies of calibration check samples. The accuracy of water methods was estimated from the data validation procedures (discussed elsewhere in this review) and the laboratory reports on the analyses of LCS samples. EPA did not estimate the accuracy of soil, sediments, and biological data because the relative matrix effects of spiked versus natural samples were not known.

Given the imprecision of the analyses as tabulated in the draft report, it is inappropriate to comment further on the presence or absence of bias.

Because EPA has not fully utilized its quality assurance data, it cannot substantiate the claim on Page 67 of the draft report:

"As a result, the Love Canal data are among the most carefully validated environmental measurements, and (given the constraints previously mentioned) are representative of the current state-of-the-art in environmental measurement methodology in terms of precision, accuracy and specificity."

VI. Data Reduction and Analysis

Scope: To determine if the data reduction and analysis are consistent with and supported by the quality of the reported data.

- A. Has evidence been presented to demonstrate the accuracy of transcription, collection and tabulation of data? If not, what additional supporting documentation is needed?

The report of the prime contractor, GCA, presents compelling evidence that sufficient care was taken in the transcription of the data and in the verification of the accuracy of the transcription and transmittal of the data into the final data base. We have no reason to believe any significant errors were introduced into the validated data base through errors in the transcription and transmittal of data.

- B. Has EPA assigned accuracy and precision limits to the reported data? Have these precision and accuracy claims been adequately justified by the quality assurance program and the available literature on the monitoring methods used? If accuracy and precision limits have not been assigned, does the documentation provided by EPA indicate that they could be assigned? If so, what documentation should EPA provide to justify precision and accuracy claims?

EPA has not incorporated precision and accuracy estimates into the validated data base.

EPA has used data generated by the quality assurance program to estimate precision and, in some cases, accuracy for the determination of particular analytes in particular media. It has been noted in several sections of this Review that EPA has not used, as fully as it could, the data generated by its quality assurance program to confirm its reported estimates of precision, accuracy, and limits of quantitation. EPA has not compared the precision and accuracy estimates derived from the quality assurance program of the Love Canal Monitoring Study to estimates derived in other studies and published in the scientific literature.

- C. Has EPA adequately interpreted the significance of "none detected" or "below detection limit"? Has EPA listed and supported detection limits for its targeted compounds? Has it supported "below detection limit" claims with documentation from its quality assurance program? Are there ways in which EPA could present the organic analytical data to

make "below detection limit" more useful or should it continue to report such data in such a manner?

EPA has provided, in its draft report, estimates of the limits of quantitation and, in some cases, limits of detection for particular analytes in particular media. Methods used to arrive at these limits are not described and should be. Wherever possible, additional data from the quality assurance program should be used to support the reported estimates. Additional comments on the limits of detection and quantitation can be found in Sections IV and V of this Appendix.

EPA has not discussed in its draft report how these limits of detection could have affected its conclusions, a central issue since most of EPA's conclusions are based on comparisons of "detects" with "non-detects." If the limits of detection are well below the level at which EPA would recommend some alternative actions to those appearing in its recommendations, then EPA should so state. If, on the other hand, the conclusions would be altered if more sensitive or more accurate data were available, EPA must consider this in its report. In any case, the conclusions drawn in the EPA report must include a statement of the concentration levels at which the conclusions are valid.

- D. What conclusions has EPA drawn from intercomparison of sample splits and sample campaigns (the collection of a number of samples from a given location over the duration of the study)? Are the conclusions justified?

The use of sample replicates has been addressed in Section V.C above. Because of the small fraction of samples containing levels of analytes above the detection limit, the conclusions drawn were limited and were used to estimate levels of precision for some of the methods and some of the sampled media. EPA has not compared variability at a given site over a period of time with analytical variability. Such comparisons may not prove to be illuminating, but would be well worth investigating.

- E. Has EPA adequately justified the rejection of data and, if not, should some data which have been rejected be reincluded in the validated data base?

The rejection of data has not always been clearly explained (see Section V.B). For data whose rejection has been clearly explained, the rather wide tolerances allowed for validation make it unlikely that acceptable data were rejected by EPA.

- F. Has EPA adequately justified the inclusion of data in the validated data base and, if not, what kinds of analyses should EPA perform on the

sample data and the quality assurance data to provide such justification?

EPA has not addressed, in its draft report, the probability of false "non-detects" in its validated data base. The GC-MS audit has given some insight into the fraction of compounds missed or misidentified at concentrations near the detection limit. Such considerations become important only when the conclusions of the study are likely to be affected. The statements and recommendations made in Section VI.C addressing the significance of limits of detection apply here as well.

- G. Has EPA incorporated precision and accuracy statements into its validated data base? If not, does it have sufficient information to do so? If it has, has it been done correctly?

Precision and accuracy statements have not been incorporated into the validated data base. Some precision and accuracy statements have been given in the appendices to the EPA draft report and those wishing to use the Love Canal data for further study must refer to these estimates when interpreting the data.

As indicated earlier in this Review, EPA should use to the fullest extent the available data to confirm these estimates of precision and accuracy.

- H. How have recovery factors been included in the validated data file? Is it clearly stated how this was done? Was the procedure justified?

Data in the validated data base have not been 'corrected' by division by recovery factors, which is consistent with current accepted practice. Recovery factors have been presented for some of the targeted compounds for the air monitoring program but not the water monitoring program. Recovery factors should be presented for all targeted compounds whenever possible. Recovery factors for the air monitoring program were calculated from performance on calibration check samples. In a similar manner, recovery factors for the water monitoring program analytes should be obtainable from laboratory control standards, and from performance evaluation and quality control samples. Recovery factors are invaluable when comparing environmental data from several laboratories.

- I. Should any of the reduced data tables be published as they now stand?

In discussions with EPA, following submission of the study template to EPA, it was determined and mutually agreed upon that this question was beyond the scope of the review requested by EPA.

VII. The EPA Audit of the GC-MS Computer Records

Scope: To determine if the EPA audit of the GC-MS computer records provides usable information on the quality of the organic analysis data and to suggest ways in which information obtained from this audit might be used.

- A. What sources of error would have and/or has such an audit uncovered? Does the audit suggest that significant numbers of such errors might be present in the validated data file? Could such errors affect substantially any conclusions which might be drawn by EPA or anyone using the data?

The EPA audit reviewed only the interpretation of GC-MS computer records (tapes) and was not a review of all the steps of the analyses conducted by the contract laboratories. EPA reviewed the computer records for 5% of the water and sediment samples. The two indices developed for the audit give a measure of the agreement between two laboratories. They do not distinguish between misidentifications and missed identifications. For example, if only one compound was present and was detected by both laboratories, but was identified incorrectly by one laboratory, the same indices would be obtained as in the situation where two compounds were present but each laboratory correctly identified only one compound and each identified a different compound. EPA gives no additional indication of the nature of the differences uncovered by the audit. For example, were differences primarily in the identification of two targeted compounds of similar chemical structure?

The use of only three comparisons between EMSC and AERL to establish the standard for agreement against which all other agreements will be compared does not seem reasonable.

These concerns aside, the GC-MS audit provided valuable insight into discrepancies in compound identification and into the effectiveness of the identification of non-targeted compounds.

The discussion on Pages VI-11 and 12 concerning the concentration levels at which disagreement occurred is convincing. No evidence is presented, however, to support the claim "the great majority of discrepancies ...[were]... in samples that were identified as heavily contaminated." Assertions concerning whether missed identifications were primarily in samples already identified as contaminated as opposed to samples considered free of contamination, if false, could influence the conclusions of the report. The seriousness of this concern would also depend on the concentration levels at which EPA is basing its conclusions and recommendations as discussed in Section VI.C above.

The EPA draft report cites as one of the major safeguards in the monitoring study, the identification in each environmental sample of the

20 most abundant non-targeted substances. Eighty samples (water, soil and sediments) were audited for the identification of non-targeted substances. In 58 of these samples, both the audit laboratory and the analytical laboratory identified no non-targeted substances. In the remaining 22 samples, the audit laboratory identified 84 non-targeted substances while the analytical laboratories found only 1. This result indicated that, for the samples audited, most laboratories did not identify non-targeted substances.

Finally, the results of the GC-MS audit of water, soil, and sediment data are not applicable to other media. An independent audit of air data is necessary if similar information is sought.

- B. Does the audit indicate there are major differences in the ways the laboratories analyzed their GC-MS tapes? How would such differences manifest themselves? Was the audit adequate to detect such differences? Should it have been?

The audit showed that laboratories differed in the identification of compounds near the detection limit. In its analysis of the audit, EPA attributed these differences to differences in computer algorithms and analysts' judgements.

The differences would manifest themselves as misidentifications or missed identifications by the analytical and/or audit laboratories. The audit appears to have been adequate to detect these differences.

- C. Should all of the GC-MS tapes corresponding to the validated data file be reanalyzed using a single program? Can we specify which program? Should GC-MS tapes corresponding to rejected data also be reanalyzed? Could faults in the analysis of the GC-MS tapes have led to incorrect rejection of data from or inclusion of data into the validated data file?

Variability introduced by use of different programs is likely to be less important than the variability introduced by the different analysts who reviewed the data.

Further audit of the GC-MS records, including review by a team of analysts using identical criteria, would appear to be necessary, so long as conclusions drawn by EPA in its Love Canal Monitoring Study are sensitive to the presence of undetected contaminants at concentrations near the detection limit.

Faults in the analysis of the GC-MS tapes are not likely to have led to incorrect rejection of data from or inclusion of data into the validated data file because the criteria for rejection were basically unrelated to possible problems with compound identifications.

APPENDIX B
DOCUMENTS RECEIVED BY NBS

| Item | Document Title | Date Received at NBS |
|---------------------------|--|-------------------------|
| <u>Bound and Titled</u> | | |
| 1. | Quality Assurance Plan Love Canal Study | 18 Aug. 81 |
| 2. | Quality Assurance Plan Love Canal Study Appendix A (Sampling Procedures) | 18 Aug. 81 |
| 3. | Quality Assurance Plan Love Canal Study Appendix B (Analytical Procedures) | 18 Aug. 81 |
| 4. | Quality Assurance Plan Love Canal Study Appendix Q (Subcontractors' QA Plans) | 18 Aug. 81 |
| 5. | Love Canal Monitoring Program GCA QA/QC Summary Report | 28 Jan. 82 |
| <u>Unbound and Titled</u> | | |
| 6. | Environmental Monitoring at Love Canal, 1980 (Designated by NBS as OEM-LC-2) | 18 Aug. 81 |
| 7. | Data and Control Charts by Level [Air]--'Four Documents' | 21 Oct. 81 |
| 8. | Audit of Gas Chromatography/Mass Spectrometry Data (GC/MS) Provided by Love Canal Project Analytical Laboratories | 21 Oct. 81 |
| 9. | GCA Report Volume I, Proposed Work Plan Love Canal Monitoring Project | 21 Oct. 81 |
| 10. | Additional EPA Love Canal Materials for NBS Review (Correspondence) | 21 Oct. 81 |
| 11. | Supplemental Documentation of EPA Love Canal Quality Assurance Program (Correspondence) | 19 Nov. 81 |
| 12. | Revised Version of Audit of Gas Chromatography/Mass Spectrometry Data Provided by Love Canal Project Analytical Laboratories (Item 8 above) | 19 Nov. 81 |

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| 13. | U.S. EPA Environmental Monitoring at Love Canal (Draft Report, Designated Copy #9) | 17 Dec. 81 |
| 14. | Appendix I List of Substances Monitored at Love Canal | 17 Dec. 81 |
| 15. | Appendix II Comparative Data and Existing Standards for Substances Monitored at Love Canal | 17 Dec. 81 |
| 16. | Revised Sections 3.3, 3.3.1, and 3.3.2 for EPA Draft Report | 28 Jan. 82 |
| 17. | Quality Assurance for Water Samples Revised Appendix III for Draft Report | 28 Jan. 82 |
| 18. | Quality Assurance for Air Samples Revised Appendix V for Draft Report | 28 Jan. 82 |
| 19. | Report on the Audit of Gas Chromatography/Mass Spectrometry Data Provided by Love Canal Project Analytical Laboratories New Appendix VI for Draft Report | 28 Jan. 82 |
| 20. | Quality Assurance Plan, Love Canal Study, Appendix Q, Revision 1 | 28 Jan. 82 |
| 21. | Quality Assurance for Soil, Sediment, and Biota Samples Revised Appendix IV for Draft Report | 17 Feb. 82 |

Unbound and Untitled

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|-----|---|------------|
| 22. | Laboratory Audits - Trip Reports Love Canal Study (Designated by NBS as OEM-LC-1) | 18 Aug. 81 |
| 23. | Love Canal Study Area: Sampling Sites (Designated by NBS as OEM-LC-3) | 18 Aug. 81 |
| 24. | Aggregate of Data from Various Sampling Areas (Designated by NBS as OEM-LC-4) | 18 Aug. 81 |
| 25. | Aggregate of Data from Various Sampling Areas (Designated by NBS as OEM-LC-5) | 18 Aug. 81 |
| 26. | Aggregate of Data from Control, Canal, and Declaration Areas (Designated by NBS as OEM-LC-6) | 18 Aug. 81 |

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| 27. | Aggregate of Data from Control, Canal, and Declaration Areas (Designated by NBS as OEM-LC-7) | 18 Aug. 81 |
| 28. | Graphical Displays of Air Monitoring Data as a Function of Sampling Date | 6 Oct. 81 |
| 29. | Quality Assurance Control Charts & Tables for Water Samples | 6 Oct. 81 |
| 30. | Quality Assurance Data (Tables) for Love Canal Soil, Sediment, and Biota | 21 Oct. 81 |
| 31. | Soil Holding Times (Internal EPA Correspondence) | 21 Oct. 81 |
| 32. | Holding Time Study for Purgeable Compounds in Water (Internal EPA Correspondence) | 21 Oct. 81 |
| 33. | Report on Audit of PJB Laboratories (Internal GCA Correspondence) | 21 Oct. 81 |
| 34. | Report on Audit of Battelle and PEDCO Laboratories (Internal GCA Correspondence) | 21 Oct. 81 |
| 35. | Collection of Public Comments Received by EPA Regarding Methods 624 and 625 and EPA Response to those Comments | 28 Jan. 82 |

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|--|--|---|---|------------------------------------|
| U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions) | | 1. PUBLICATION OR REPORT NO. NBSIR 82-2511 | 2. Performing Organ. Report No. | 3. Publication Date May 1982 |
| 4. TITLE AND SUBTITLE | | | | |
| 5. AUTHOR(S) | | | | |
| 6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions) NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234 | | | 7. Contract/Grant No. | 8. Type of Report & Period Covered |
| 9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP) U.S. Environmental Protection Agency 401 M Street, SW Washington, D.C. 20460 | | | | |
| 10. SUPPLEMENTARY NOTES | | | | |
| <input type="checkbox"/> Document describes a computer program; SF-185, FIPS Software Summary, is attached. | | | | |
| 11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) ABSTRACT This report is a review conducted by the National Bureau of Standards at the request of the U.S. Environmental Protection Agency (EPA) of the analysis for organic chemicals conducted by EPA in the Love Canal Area of Niagara Falls, New York. | | | | |
| 12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) Love Canal, Monitoring, Organic Analysis, Environment, Pollution | | | | |
| 13. AVAILABILITY <input type="checkbox"/> Unlimited <input checked="" type="checkbox"/> For Official Distribution. Do Not Release to NTIS <input type="checkbox"/> Order From Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402. <input type="checkbox"/> Order From National Technical Information Service (NTIS), Springfield, VA. 22161 | | | 14. NO. OF PRINTED PAGES 15. Price | |

